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TENT COOPERATION TREAT

### **PCT**

C'D 20 JAN 2005

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### INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference GIP17PT03			file reference	FOR FURTHER ACTION  See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)					
International application No. PCT/EP 03/50909				International filing dat 28.11.2003	e (day/moni		Priority date (day/month)		
International Patent Classification (IPC) or both national classification and IPC C08G18/48									
Applicant  LAMBERTI SPA et al.									
1.	. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.								
2.	This REPORT consists of a total of 4 sheets, including this cover sheet.								
	This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).								
i	The		s consist of a total of		auve msuu	ictions ander (r	ie PC1).		
3.	This	report con	itains indications rela	ating to the following	items:				
	 	☐ Bas	sis of the opinion						
	Ш		•	pinion with regard to	novelty, in	ventive step an	nd industrial applicability	ı	
	IV V	⊔ Lac	k of unity of invention	n					
	V	□ Rea     citat	isoned statement un tions and explanatio	der Rule 66.2(a)(ii) w ns supporting such si	rith regard atement	to novelty, inve	entive step or industrial	applicability;	
	VI	☐ Cer	tain documents cited	1					
	VIII VIII			ternational application					
	VIII	Li Cen	tain observations on	the international app	lication				
Date of submission of the demand					Date of co	ompletion of this	report		
30.06.2004					19.01.2	005			
Name and mailing address of the international preliminary examining authority:					Authorize	d Officer		nat Poten	
European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465				_	Rebollo, J e No. +49 89 239	99-8670			



## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/EP 03/50909

ł.	Basis	of the	report
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1. With regard to the **elements** of the international application (Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)):

	De	Description, Pages							
	1-2	20	as originally filed						
	Cla	aims, Numbers							
	1-1	17	received on 24.09.2004 with letter of 16.09.2004						
2.	. Wi lan	With regard to the language, all the elements marked above were available or furnished to this Authority is language in which the international application was filed, unless otherwise indicated under this item.							
		These elements were available or furnished to this Authority in the following language: , which is:							
		the language of a tr	anslation furnished for the purposes of the international search (under Rule 23.1(b)).						
	the language of publication of the international application (under Rule 48.3(b)).								
		the language of a tra Rule 55.2 and/or 55	anslation furnished for the purposes of international and including						
3.	Wit inte	With regard to any <b>nucleotide and/or amino acid sequence</b> disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:							
		contained in the international application in written form.							
		furnished subsequently to this Authority in computer readable form.							
		The statement that t listing has been furn	he information recorded in computer readable form is identical to the written sequence ished.						
4.	The	e amendments have resulted in the cancellation of:							
		the description,	pages:						
		the claims,	Nos.:						
		the drawings,	sheets:						
5.		This report has been been considered to g	established as if (some of) the amendments had not been made, since they have go beyond the disclosure as filed (Rule 70.2(c)).						
		(Any replacement sh report.)	eet containing such amendments must be referred to under item 1 and annexed to this						
6.	Add	dditional observations, if necessary:							

Form PCT/PEA/409 (January 2004)



# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/EP 03/50909

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)

Yes: Claims

No: Claims

Inventive step (IS)

Yes: Claims

1-17

1-17

No: Claims

Industrial applicability (IA)

- Yes: Claims No: Claims

1-17

2. Citations and explanations

see separate sheet



## INTERNATIONAL PRELIMINARY

International application No. PCT/EP 03/50909

#### **EXAMINATION REPORT - SEPARATE SHEET**

#### Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

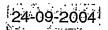
Reference is made to the following documents:

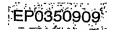
D1: EP-A-0 060 430 (GOLDSCHMIDT AG TH) 22 September 1982 (1982-09-22)

D2: WO 99 52961 A (CLARIANT INT LTD ; DANNER BERNARD (FR): CLARIANT FINANCE BVI LTD (V) 21 October 1999 (1999-10-21) cited in the application

D3: EP-A-1 184 400 (BAYER AG) 6 March 2002 (2002-03-06)

The subject-matter of claims 1 to 17 of the present application appears to be novel (Article 33(2) PCT) and involve an inventive step (Article 33(3) PCT) because the skilled person trying to ascertain the teaching of D1, where (implicitly aqueous) dispersions of non-ionic blocked polyisocyanates obtained from the reaction of a polyisocyanate, a blocking agent and a non-ionic diol having poly(ethylene and/or polypropylene ether) side-chains were disclosed (see passages cited in the search report) would not have obtained from this document any indication to use a thermally reversible isocyanate blocking agent (disclosed in D2 or D3), as the only isocyanate blocking agents disclosed in D1 (see page 6, lines 35 to 38) are thermally stable, in order to solve the problem underlying the invention: to provide stable aqueous dispersions of non-ionic block polyisocyanates.





#### **CLAIMS**

- 1. Aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates obtained from the reaction of:
- (i) a polyisocyanate;
- 5 (ii) a thermally de-blockable -N=C=O blocking agent;
  - (iii) a non-ionic alkoxylated diol having general formula I:

$$R_1CH_2O-(-CH_2CH_2O)n-(CH_2CHCH_3O)m-R_2$$
(I)

wherein:

$$R_1 = HO$$
OH

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15

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(II)

or

$$R_1 = R_3$$
HO (III)

 $R_2$  and  $R_3$  are equal or different and are chosen among methyl, ethyl, n-propyl, i-propyl, n-butyl, i-butyl; n is a number from 0 to 40; m is a number from 0 to 40; n + m is a number from 20 to 80.

- 2. Aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates according to claim 1., wherein n + m is a number from 20 to 40.
- 3. Aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates according to claim 1. or 2., wherein the non-ionic alkoxylated diol (iii) has the general formula I:

$$R_1CH_2O-(-CH_2CH_2O)n-(CH_2CHCH_3O)m-R_2$$
(I)

wherein:

$$R_{\tilde{1}} = R_{\tilde{3}}$$
HO

(III)

- $R_2$  is methyl,  $R_3$  is ethyl, n is a number from 15 to 30 and m is a number from 0 to 10.
  - 4. Aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates according to any of the preceding claims, wherein the polyisocyanate (i) is the isocyanurate obtained from 1,6-hexamethylenediisocyanate and the reaction product of trimethylol propane and toluenediisocyanate (its isomers 2,4 and 2,6 being in a weight ratio of 80:20).
  - 5. Aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates according to any of the preceding claims, wherein the blocking agent (ii) is 3,5-dimethylpyrazole.
- 6. Process for the preparation of aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates comprising the following steps:
  - a. a polyisocyanate (i) and a non-ionic alkoxylated diol (iii) of the general

 $R_1CH_2O-(-CH_2CH_2O)n-(CH_2CHCH_3O)m-R_2$ 

formula

20

10

(I)

wherein '

$$R_1 = HO$$
 OH

(II)

5

15

25

or

$$R_1 = R_3$$
HO

(III)

 $R_2$  and  $R_3$  are equal or different and are chosen among methyl, ethyl, n-propyl, i-propyl, n-butyl, i-butyl;

n is a number from 0 to 40;

m is a number from 0 to 40;

n + m is a number from 20 to 80,

are reacted at a temperature of 30°-120°C, their equivalent ratio being such that the percentage of free isocyanate groups in the resulting oligomer is from 3 to 10 and the percentage in weight of ethoxyl groups is from 10 to 40%;

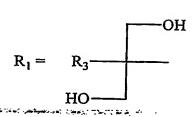
- b. the thus obtained oligomer is reacted with an amount of a blocking agent (ii) such that the equivalent ratio of the isocyanate groups of the oligomer and the blocking agent (ii) is from 1:0.98 to 1:1.30;
- c. the thus obtained mixture is dispersed into water under vigorous stirring to obtain a dispersion having a solid content of from 20 to 40% by weight.
  - 7. Process for the preparation of aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates according to claim 6., wherein the non-ionic alkoxylated diols (iii) have the general formula I:

R<sub>1</sub>CH<sub>2</sub>O-(-CH<sub>2</sub>CH<sub>2</sub>O)n-(CH<sub>2</sub>CHCH<sub>3</sub>O)m-R<sub>2</sub>

**(I)** 

wherein:

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(III)

 $R_2$  is methyl,  $R_3$  is ethyl, n is a number from 15 to 30 and m is a number from 0 to 10.

- 8. Process for the preparation of aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates according to claim 6., or 7., wherein the polyisocyanate (i) is the isocyanurate obtained from 1,6-hexamethylenediisocyanate and the reaction product of trimethylol propane and toluenediisocyanate (its isomers 2,4 and 2,6 being in a weight ratio of 80:20).
  - 9. Process for the preparation of aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates according to claim 6., 7., or 8., wherein the step b. is preceded by dilution of the reaction mixture obtained in a. with from 0.10 to 0.50 parts by weight of a water mixable polar solvent.
  - 10.Process for the preparation of aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates according to claim 9., wherein the water mixable polar solvent is chosen among methyl ethyl ketone, acetone, cyclohexanone.
- 20 11. Process for the preparation of aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates according to any of the claims from 6. to 10., wherein in step a. the equivalent ratio of polyisocyanate (i) and alkoxylated diol (iii) is such that the percentage in weight of ethoxyl groups is from 20 to 30%.
- 25 12.Process for the preparation of aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates according to any of the claims from 6. to 11., wherein the blocking agent (ii) is chosen from butanone oxime and 3,5-dimethylpyrazole.

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- 13.Process for the preparation of aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates according to any of the claims from 6. to 12., wherein the amount of blocking agent (ii) is such that the equivalent ratio of the isocyanate groups of the oligomer and the blocking agent (ii)
- 14.Process for the preparation of aqueous dispersions of non-ionic -N=C=O blocked polyisocyanates according to any of the claims from 6. to 13., wherein in step c. the mixture is dispersed into water under vigorous stirring to obtain a dispersion having a solid content of from 25 to 35% by weight.

"Is from 1:1 to 1:1:2: ---

- 15.Procedure for the oil- and/or water-repellent finishing of textiles, characterised by the fact that, as finishing agent, an aqueous composition is used, said aqueous composition comprising at least an organic perfluorinated polymeric compounds and from 0.1 to 10% by weight, on the total weight of the composition, of an aqueous dispersion of non-ionic -N=C=O blocked polyisocyanates according to any of claims from 1. to 5., the weight ratio between the solid fraction of the aqueous dispersion and the perfluorinated polymeric organic compounds being comprised between 1:1 and 1:15.
- 20 16.Textile printing pastes characterised by the fact that they contain from 0.3 to 5% by weight of an aqueous dispersion according to any of claims from 1. to 5.
  - 17.Textile printing pastes characterised by the fact that they contain from 1 to 3.5% by weight of an aqueous dispersion according to any of claims from 1. to 5.